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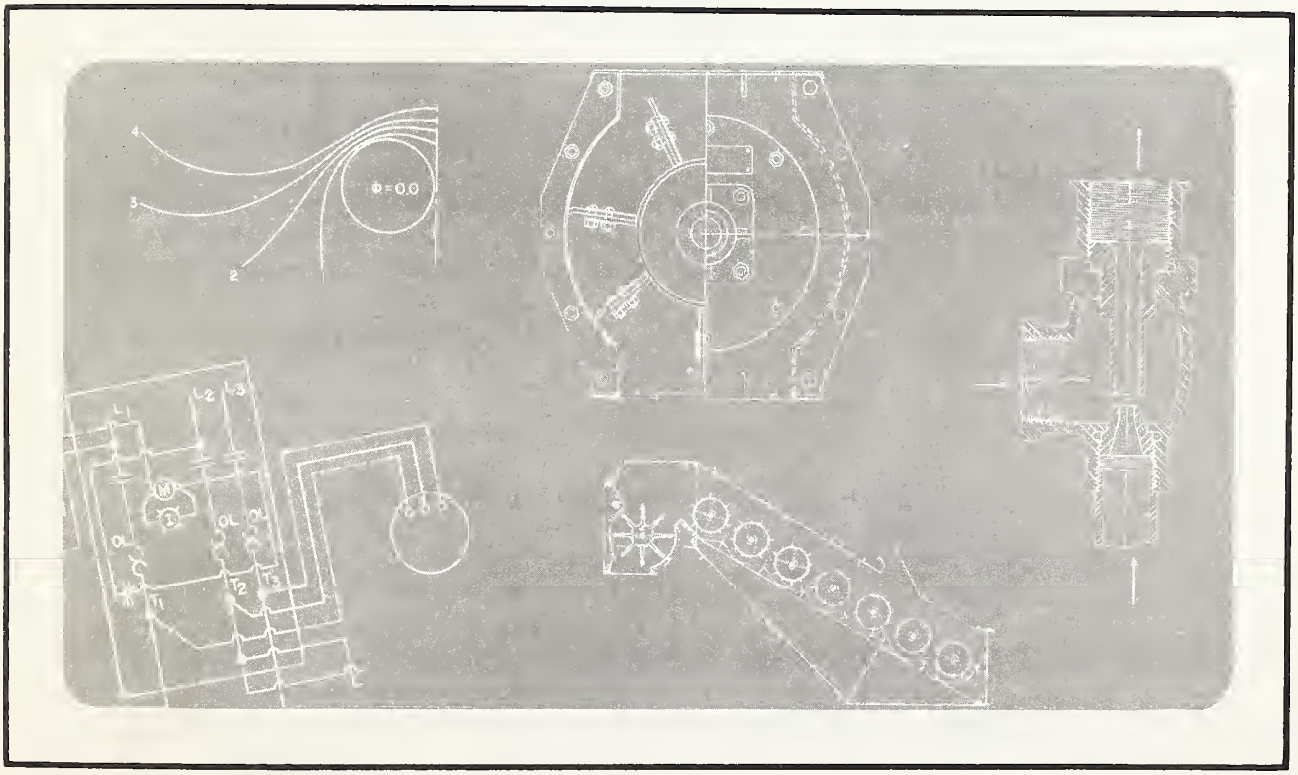
In-Transit Fumigation of Truck-Ship Containers With Hydrogen Phosphide

A Feasibility Study



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In-Transit Fumigation of Truck-Ship Containers With Hydrogen Phosphide

A Feasibility Study

By Edward Jay,[1] Robert Davis,[1] and James M. Zehner[2]

ABSTRACT

Truck-ship containers in three sizes (20, 35, and 40 ft) were fumigated loaded or empty, stationary or in transit. The three hydrogen phosphide sources used extensively in the studies controlled test insects effectively and left residues that were generally below tolerance levels set by the U.S. Environmental Protection Agency. Besides bioassays and residue analyses, studies were done on container sealing, aeration, and simulated accidents. The resulting data show that, under the conditions prevalent during the tests, in-transit fumigation of these containers can be done safely and effectively. Index terms: chemical control (insects), containers, freight transportation, fumigation procedures, hydrogen phosphide, insect control, phosphine, stored-product pests.

INTRODUCTION

Many centers for processing, warehousing, and distributing food are involved in shipping subsistence commodities by truck-ship containers to overseas markets. Because of the length of the marketing or distribution systems resulting from extended warehousing and transportation, these commodities often become infested with insects. Currently, the most common method of dealing with insect infestation in containers is to fumigate both the container and its contents. These fumigations are time consuming and expensive. Therefore, they can cause significant problems in meeting shipping and other distribution schedules.

The most common fumigation procedure in current practice is to place the container in a secure location, cover it with a tarp or a plastic film, and fumigate it with hydrogen phosphide gas. Depending on the temperature, these fumigation periods can vary from 3 to 10 days that must be followed by appropriate aeration periods. One way to save time and money would be to effect these fumigations in transit when needed or required for quarantine. In 1977, the U.S. Department of Defense's Armed Forces Pest Management Board recommended that the feasibility of such in-transit fumigations be investigated for use in shipping military subsistence to overseas installations. The Board at the same time requested permission from the U.S. Environmental Protection Agency (EPA) to conduct this research. EPA approved the initiation of this research if the following conditions were met: All containers had to carry a placard with the skull and crossbones, the words "poison gas," a statement of the hazards, the name of the fumigant used, and the dates of fumigation. EPA also ad-

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vised that the program had to meet the following conditions:

1. The program had to be carried out under strict control of the U.S. Departments of Defense and Agriculture.
2. The program had to be limited in size. EPA authorized only enough fumigant to treat those containers that would be continually monitored from fumigation to unloading.
3. Containers had to be accompanied and monitored during fumigation, shipment, and unloading.
4. Concentrations of fumigant gas in the cargo area had to be monitored during transit and unloading.
5. There had to be continual monitoring for fumigant gas in the cab and around the truck.
6. Residue samples had to be taken before unloading. Grain with residues above the established tolerance had to be destroyed or held until residue levels dropped to acceptable levels.
7. Types of containers to be used had to be described.
8. Evidence had to be submitted that an explosion would not occur if a vehicle that had been fumigated were involved in an accident.

All preliminary and static (stationary) phases of the study were done in a secure location at the U.S. Agricultural Research Service's Stored-Product Insects Research and Development Laboratory at Savannah, Ga. The in-transit phase was done over varying distances on U.S. Highway 17 south of Savannah. All in-transit tests were done with a chase car in place to provide emergency services in the event of an accident, and a statement of the hazards involved was carried with the container when it was in transit.

This report presents the results of the study. We believe that the report has adequate information for development of safe and effective procedures for in-transit fumigation of truck-ship containers with hydrogen phosphide.

METHODS AND MATERIALS

Fumigant

Hydrogen phosphide (PH_3), or phosphine, is an efficient fumigant that is released from aluminum phosphide and magnesium phosphide on contact with water (as moisture in air, for example). Hydrogen phosphide has a specific gravity of 1.2; has a carbide, or garlicky, odor; and will explode at concentrations of over 1.79% or 17,900 p/m (parts per million) in air. Based on an 8-hour workday and 40-hour workweek, a TLV (threshold level value) of 0.3 p/m has been established by the National Conference of Industrial and Government Hygenists for workers exposed to this fumigant. EPA has established a residue tolerance on raw commodities of 0.1 p/m and on processed commodities of 0.01 p/m. Some other characteristics of this fumigant are:

1. Very active when compared with other fumigant gases.
2. Penetrates the commodity and is aerated from it rapidly.
3. Does not react with fumigated products to form a permanent residue.
4. Requires longer exposure periods (3–5 days) than do other fumigants to obtain complete control of insects.
5. Concentrations of 100 to 700 p/m are toxic to insects in the 3- to 5-day period, but lower concentrations will give control over longer time periods.
6. Reacts with some metals, particularly with copper.

Four sources of hydrogen phosphide were used in these studies:

1. Phostoxin pellets (Degesch m.b.H., Frankfurt am Main, West Germany). These are small, compressed, 0.6-g pellets of aluminum phosphide mixed with ammonium carbamate. On exposure to moisture in air, the pellets decompose slowly to yield hydrogen phosphide, aluminum hydroxide, ammonia, and carbon dioxide.
2. Phostoxin Prepac (Degesch m.b.H., Frankfurt am Main, West Germany). These are packaged Phostoxin pellets as described above. Each Prepac contains 165 pellets, and this formulation is approved by EPA for use on raw and processed agricultural commodities.
3. Degesch Fumi-Cel (Degesch m.b.H., Frankfurt am Main, West Germany). This formulation comes as a unit of magnesium phosphide impregnated into a 10¼- by 7- by ¼-inch polymer plate. The magnesium phosphide gives off hydrogen phosphide faster than does the aluminum phosphide formulation and does

not produce ammonia, which may damage fresh fruit during fumigation.

4. Detia Gas Ex–B (Detia Export G.m.b.H., Laudenbach, West Germany). This formulation, aluminum phosphide plus a stearate binder, gives off hydrogen phosphide without ammonia production. The formulation used is supplied in 34-g bags that, when exposed to moisture, give off the gas.

Containers

The containers used in these studies were typical of those available in the commercial market. We used two 40-ft (2,337-ft^3) containers for the initial phases of the study. These were made of heavy sheet metal and had tongue-and-groove wooden floors. We also used a 35-ft (2,150ft^3) aluminum container to study the value of a polyethylene curtain installed in front of the doors. This container also had a tongue-and-groove wooden floor. We used a 20-ft (1,280-ft^3) container during the later stages of the study. This container was similar in construction to the 40-ft containers and was used for most of the in-transit portions of the study.

Container Sealing and Pressure Testing

The studies to find adequate sealing materials and a usable method of pressure testing were conducted in the two 40-ft containers. Some preliminary evaluations of sealants were conducted with 2-inch boards nailed tightly together to simulate the wood tongue-and-groove container flooring. Thirteen compounds were tested for their suitability (see appendix).

To establish a method of pressure testing containers to assess their suitability for retaining fumigants, we used duct tape on all the floorboard junctions and a commercial aluminum roofing compound at the floor-to-wall junctions on one 40-ft container. The rear doors were sealed with 2-inch duct tape. In the first series of pressure tests, we cut two holes in the wooden floor of a 40-ft container. Into one of these holes, we inserted the hose from the pressure side of a shop vacuum cleaner for pressure testing. The other hole was for insertion of polyethylene lines to measure pressure buildup and its rate of decay. In each test, two daytime-orange smoke flares were set off in the container, and the doors were closed and sealed. The vacuum was then used to pressurize the container, forcing the orange smoke out of remaining holes to show areas needing additional sealing. In a second series of tests, we pressurized containers and recorded the rate of decay.

Fumigation Tests With 35- and 40-Ft Containers

Several tests, involving both static and in-transit fumigations of empty containers, were conducted in the two 40-ft containers. One container was prepared as above with duct tape and roofing compound. In an effort to minimize costs, we covered the floor of the other container with a 6-mil sheet of polyethylene. We used sheets of cardboard on the film to protect it from the forklift that would normally be used in loading. The doors on both containers were sealed with duct tape and duct putty around the hinges and other large openings. The fumigant hydrogen phosphide was generated from Phostoxin pellets placed in screened boxes next to the container doors. The dosage, 380 pellets, was based on the label, which recommends the use of 165 to 200 pellets per 1,000 ft^3. In the empty in-transit tests, the containers were held for 24 hours and then hooked up to a tractor and pulled over the highway. Concentrations of hydrogen phosphide were measured regularly throughout the tests in both the stationary and in-transit containers and in the tractor cabs. Also, gas samples were taken around the containers during the fumigation periods. All gas samples in all tests were taken with an MSA universal sampling pump No. 83499 (Mine Safety Appliances, Pittsburg, Pa.) and Dräger tubes, either phosphine 50/a. or phosphine 0.1/a. (Drägerwerk A.G., Lubeck, West Germany). In all tests, we placed two thermistors inside the container to measure temperature.

We conducted four static fumigation tests with a loaded 40-ft container. The container was prepared as described above with 6-mil polyethylene sheeting and cardboard. It was then loaded with pallets holding about 5,000 lb of flour, 1,800 lb of soybeans, 1,800 lb of corn, and 240 cardboard boxes containing in-shell peanuts (about 4,700 lb). Four pallets containing boxed peanuts and packages or boxes containing flour, cornmeal, noodles, macaroni, rice, or dry cereal were placed at the front, middle, and rear of the container to

serve as bioassay sites. The container's total volume was about one-half filled by these commodities. Cages containing either red flour beetle, *Tribolium castaneum* (Herbst), larvae or pupae; black carpet bettle, *Attagenus megatoma* (Fabricius) larvae; or eggs or larvae of the almond moth, *Ephestia cautella* (Walker), were placed inside the boxes or bags as bioassays at each site. One bioassay site (pallet) was similarly set up in an adjacent 35-ft container, and insects were placed in the commodity in this container to serve as controls. Gas-sampling lines were run to each of the four bioassay sites. We then fumigated the container by placing two Phostoxin Prepacs containing 165 pellets each in the rear of the container. The 330 pellets were the recommended dosage for treating 2,000 ft^3 with the Prepacs. The rear doors were then closed, and the areas around the gaskets were sealed with duct tape and putty.

We ran three more static fumigation tests in this loaded 40-ft container, basically to determine if the 3- and 4-day static fumigations currently conducted at the U.S. Department of Defense's depot at Mechanicsburg, Pa., were effective. Several species and life stages of stored-product insects were placed in the commodity at four locations in the container. In each of these tests, two Phostoxin Prepacs were taped to the cardboard covering the floor in the rear of the container just inside the doors. The doors were then closed and sealed with duct tape and putty.

We used the 35-ft container for two tests in an effort to assess the value of a polyethylene curtain inside the doors. This curtain was held in place with a metal track just inside the container doors. The track-and-curtain system (Trans Fresh Corp., Salinas, Calif.) was designed to prevent leakage of cold air from around the doors of refrigerated containers and takes two people about 15 minutes to install. The floor of this container was covered with a taped-down polyethylene sheet, but the doors were not sealed with duct tape. The container was fumigated twice for periods of 7 days each with 330 Phostoxin pellets. One fumigation was conducted without a curtain and the second with the curtain installed.

The 35-ft container was also used for in-transit fumigation studies. The empty container was pulled in two separate trips of about 60 miles in about 1.5 hours of onroad time per trip. The floor of the container was sealed with polyethylene film overlaid with cardboard, and gas-sampling lines were run from the container into the tractor cab to allow continuous, in-transit gas sampling. Samples were also taken hourly in the tractor cab.

Fumigation Tests With a Loaded 20-Ft Container

The fumigation of a loaded 20-ft container was replicated 13 times; 3 replications were static, 10 were in transit. In the static tests, we prepared the 20-ft container by placing 6-mil polyethylene and cardboard on the floor and then loaded it with 11,920 lb of material on pallets. The load was made up of food items such as noodles, rice, flour, and cornmeal and of miscellaneous supplies such as paper towels, toilet tissue, drinking cups, desk trays, paper napkins, and metal chairs. Five gas-sampling lines were installed in the container, and four of these were placed in boxes containing commodities, while the fifth sampling line was located in the free airspace. Two sites were selected in the container for bioassay and residue studies, one in an upper pallet in the front and one in a lower pallet in the rear. Before the in-transit tests, we ran static tests on the container to establish a decay-rate baseline for the in-transit fumigations.

The static and in-transit fumigation tests were first run with three bags of Detia Gas Ex–B per container. The recommended dosage for 1,000 ft^3 with this formulation is three to six bags. Similarly, we ran static tests with one Degesch Fumi-Cel plate per container (recommended dosage: one plate per 1,000 to 1,600 ft^3) and with Phostoxin Prepacs (one Prepac per container) before beginning in-transit tests for each formulation. All hydrogen phosphide sources were taped to the cardboard liner at the rear of the container just inside the doors.

During the in-transit tests, the gas-sampling lines were run into the tractor cab to allow continuous sampling of the atmosphere in the container. Gas samples were also taken periodically with low-level detection tubes in the tractor cab. During each in-transit trip, the tractor and trailer holding the container were parked for periods of about ½ to 1¼ hours to simulate a coffeebreak or meal. During these periods, the tractor cab was closed, and a gas-sampling line was installed in the cab. At the end of the break, the cab was sampled for hydrogen phosphide with low-level detection tubes. Also during the breaks, we took gas samples around the container with low-level

tubes, particularly around the doors, to determine if detectable amounts of hydrogen phosphide were present.

Bioassays

Bioassays were conducted in loaded 40- and 20-ft containers. Three cages of each species and life stage of insects, along with appropriate rearing media, were placed in a commodity (usually flour) next to a gas-sampling line in the container. Controls were held in a commodity in a container next to the treated container. In the 40-ft container, four static bioassay tests were conducted. In the first, we made a 3-day exposure at four bioassay sites with larvae of the almond moth (18 days old), the red flour beetle (24 days old), and the black carpet beetle (larvae that rode on a 12-mesh sieve—U.S. Standard Sieve Screen Series No. 14—during sieving). We ran two more tests of 91 and 95 hours at the same fumigant doses and with the same test insects. We made the fourth bioassay exposure for 7 days at the same four sites with eggs of the almond moth and the red flour beetle, larvae of the black carpet beetle, pupae of the almond moth, and 1- to 7-day-old adults of the red flour beetle.

In the 20-ft container, bioassays were conducted as in the 40-ft containers. We used red flour beetle larvae (17 to 25 days old) and black carpet beetle larvae (selected by taking those that rode a No. 14 sieve). Thirteen 4-day exposures were made in this manner, 10 while the container was on the road, and 3 while it remained static.

Accident Simulations

We recognized that an accident could happen to a fumigated container in transit. But accidents are hard to simulate since no two would be alike. We assumed, however, that the most fragile point on a container, and the point most likely to rupture on impact, would be the doors. So we threw the container doors open and sampled for hydrogen phosphide in their immediate vicinity. We did several of these accident simulations to test various different fumigant concentrations. For each simulation, we taped the formulation that the fumigant was derived from to the floor in the rear of the container immediately inside the doors.

We did three of these simulations with an empty 40-ft container and three with one that was loaded. Gas samples were taken outside the container, 1 and 4 ft from the rear of the open doors and 5 ft down the side of the container from the doors. Five similar studies were also conducted with the empty 35-ft container with and without the polyethylene curtain in place. With a loaded 20-ft container, we did nine accident simulation studies 4 days after fumigation began in a manner similar to those run with the 40- and 35-ft containers. These gas samples were taken only at 1 and 3 ft from the rear of the open trailer doors, and none were taken at the side of the trailer.

Aeration Studies and Residue Analyses

In most cases, we aerated the container at the end of a fumigation by opening the doors and removing the fumigant source. We allowed the container to air out for 3.5 to 4 hours and then sampled the atmosphere. During each of the 10 in-transit and 3 static fumigations of the loaded 20-ft container, we took commodity samples for hydrogen phosphide residue analysis. We chose two sites in the container, one high in the front and the second low in the rear next to the fumigant source. Before each fumigation, we prepared two boxes, each containing one package of the following: 8 oz of cornflakes, 5 lb of wheat flour, 5 lb of corn grits, 3 lb of rice, and 5 lb of cornmeal. We also put two 12-oz packages of blackeyed peas in each box. The boxes were then placed in the container at the two sites. An additional box was prepared in the same manner and held in a warehouse to serve as a control. At the conclusion of the fumigation, we removed the boxes from the container and the warehouse and sampled them in duplicate for residue analysis. We used Rosebrook's (1972) method for residue analysis.

RESULTS AND DISCUSSION

Container Sealing and Pressure Testing

The tongue-and-groove floors were found not to be gastight. The doors, although rubber-

gasketed, were also potential areas for gas leaks. The 20- and 40-ft containers had four vents in the inner four top corners to relieve pressure in air transport. These vents were obvious leak points and were taped with duct tape in all tests conducted in all sheet-metal containers.

Most of the sealing compounds tested possessed too low a viscosity, and they ran through the cracks (see appendix). The obvious solution is to calk the cracks to make them gastight, but we believe that this procedure would require too much labor for the proposed use of these containers. Also, container companies may not wish to have their equipment altered significantly. Costs and labor for the container that had its floor sealed with duct tape for pressure tests were about $12 (1977) for duct tape and about 2 man-hours of labor. The duct tape could be removed easily after the container was emptied. We applied pressure to the container sealed in this manner with the vacuum cleaner and noted how long it took the pressure to decay. In one test, we applied pressure equal to 3 inches of H_2O to the container, and the pressure decayed to 2 inches of H_2O in 37 minutes and to 1 inch of H_2O in 105 minutes, indicating a relatively tight seal. In another test, pressure decay from 2.6 inches of H_2O to 1.0 inch of H_2O took 90 minutes.

Table 1.—Concentrations of PH_3 in an empty 40-ft container at various stages during transit[1]

Miles driven	Concentration: Average[2] (p/m)	Concentration: Range (p/m)
0	627	530–690
3	655	575–690
17	541	400–650
31	518	475–550
42	483	450–510
58	295	250–325
78	358	275–450

[1]Joints in floor and rear door were sealed with duct tape. Transit began 24 hours after start of fumigation. Average speed was 30.7 mi/h. Ambient temperature was about 90° F; container temperatures ranged from 99° to 105° F during the trip.

[2]Average from 4 sampling lines at different locations in the container.

Fumigation Tests With 35- and 40-Ft Containers

A static fumigation of the tightly sealed (duct tape on floors) 40-ft empty container was conducted at ambient temperatures of 63° to 70° F. After 36 hours, hydrogen phosphide concentrations were >550 p/m. We stopped the test at this point, since we determined that the container was sealed too tightly for standard military operations.

The fumigation of the 40-ft empty container sealed with polyethylene on the floor and duct tape on the doors was conducted at ambient temperatures ranging from 79° to 96° F, and temperature inside the container ranged from 83° to 123° F during the tests. Twenty-four hours after the container was sealed, the average concentration was >485 p/m; the maximum observed concentration was >670 p/m after 27 hours. It was >140 p/m 160 hours after fumigation began. The 6-mil polyethylene and the door sealing seemed to hold the hydrogen phosphide in the container satisfactorily. So we used these rapid, inexpensive sealing techniques in all instances for the

Table 2.—Concentrations of PH_3 in a loaded 40-ft container at various times after start of static fumigation[1]

Hours after PH_3 introduction	Concentration[2] (p/m): Test 1[3]	Test 2[4]	Test 3[5]
17	[6]3.5	159	88
23	[6]51	216	170
26	[6]38	222	243
43	248	133	316
47	310	155	328
50	296	137	377
67	266	268	319
71	318	356	318
74	306	299	......
91	200	216	......
95	237	......	......

[1]Floors covered with 6-mil polyethylene and cardboard. Doors sealed with duct tape.

[2]Average from 4 sampling lines at different locations in container.

[3]Range of ambient temperatures: 63°–86° F; range of temperatures in container: 60°–70° F.

[4]Range of ambient temperatures: 65°–86° F; range of temperatures in container: 57°–69° F.

[5]Range of ambient temperatures: 61°–77° F; range of temperatures in container: 62°–66° F.

[6]Lines blocked; cleared by having air blown back through them.

duration of the container tests, except for the tests in the 35-ft container.

In one in-transit fumigation of an empty 40-ft container tightly sealed as described above with duct tape on the floor, the hydrogen phosphide concentration, which averaged 627 p/m at the start of the road trip, dropped to an average of 358 p/m at the end (table 1). So leakage did occur. Hydrogen phosphide was not detected in the cab while the truck was moving. The tractor and container were stopped for 28 minutes to simulate a coffeebreak, and the windows on the tractor were closed for 23 minutes after the unit stopped. Gas samples from the cab of the tractor taken after the 23 minutes revealed that no hydrogen phosphide was present. Container concentrations 16.5 hours after the end of the trip averaged 225 p/m (range: 200 to 250 p/m). Thirty-six hours after the end of the trip, concentrations averaged 160 p/m.

In one static fumigation of the loaded 40-ft container with the floor covered with polyethylene film, we observed an average concentration of 67 p/m of hydrogen phosphide (range: 50 to 250 p/m) 1 day after treatment; 2 days after treatment, the concentration averaged 233 p/m (range: 120 to 350 p/m); 3 days after treatment, the average concentration was 198 p/m (range: 50 to 300 p/m). Six days after treatment, the average concentration was 7 p/m (range: 5 to 10 p/m). The temperature range during the test was 74° to 101° F; temperature in the bioassay commodity ranged from 82° to 84° F. (See table 2 for conditions and results of the three other tests with the loaded 40-ft container.)

Table 3.—Concentrations of PH_3 related to use of a polyethylene curtain inside container doors; container static during fumigation[1]

Days after PH_3 introduction	Concentration (p/m)			
	Without curtain		With~~out~~ curtain	
	Average[2]	Range	Average[2]	Range
1	295	60–490	544	500–600
2	278	100–430	524	300–700
3	226	110–300	290	150–390
4	90	70–100	99	60–175
7	7.5	0–20	42.5	35–50

[1]35-ft empty container.

[2]Average from 4 sampling lines at different locations in container.

The static fumigation of the 35-ft container with the curtain gave consistently higher concentrations inside the container than did the fumigation of the container without the curtain (table 3). On the second day after fumigation, the container with the curtain had an average concentration of 524 p/m of hydrogen phosphide, while the container without the curtain had an average concentration of 278 p/m. This test showed the high amount of leakage occurring around the rear door, since most other variables in the two treatments (including temperature, which ranged from 76° to 112° F inside the container) were similar. Since the doors were not sealed with duct tape, these differences were greater than they would have been had the doors been sealed.

In in-transit fumigation of this container, concentrations of hydrogen phosphide before travel were higher in the container without the curtain (table 4). During the onroad period, concentrations were generally higher in the container with the curtain. Since the fumigant was still evolving during these tests, it is possible that we might have detected greater differences between them if the containers had not been moved until 48 or 72 hours after the start of the fumigations.

Table 4.—Concentrations of PH_3 related to use of a polyethylene curtain inside container doors; container in transit for part of fumigation[1]

Hours after PH_3 introduction	Concentration[2] (p/m)		Remarks
	With curtain	Without curtain	
5	103	155	Static.
22	193	258	Static.
26	384	289	Static.
27	381	300	On road.
27.25	394	356	On road.
27.5	413	325	On road.
[3]28.75	309	263	On road.
29	350	333	On road.
29.25	350	242	On road.
46	331	306	After parked overnight.

[1]35-ft empty container.

[2]Average from 5 sampling lines at different locations in container.

[3]Between 27.5 and 28.75 hours was a break when the container was parked.

Table 5.—Concentrations of PH_3 (from each of 3 sources) in a loaded 20-ft container at various times after start of static fumigation[1]

Hours after PH_3 introduction	Concentration[2] (p/m) produced by—		
	Detia Gas Ex-B	Degesch Fumi-Cel plates	Phostoxin Prepacs[3]
4	45	82	51
24	308	390	181
48	348	411	85
72	308	278	43
96	86	134	30

[1]Temperature in the container ranged from 77° to 104° F in the Detia Gas Ex-B test, from 73° to 116° F in the Degesch Fumi-Cel plate test, and from 72° to 106° F in the Phostoxin Prepac test.

[2]Average from 5 sampling lines at different locations in container.

[3]Very windy during test.

Fumigation Tests With a Loaded 20-Ft Container

In tests on the loaded 20-ft container (table 5), a static fumigation with the Detia Gas Ex-B bags produced a concentration average of 308 p/m after 24 hours; in 48 hours, it averaged 348 p/m. The hydrogen phosphide concentration averaged 308 p/m after 72 hours and dropped to 86 p/m after 96 hours. Distribution of these concentrations throughout the container was generally good during the test. A similar buildup and decay of the hydrogen phosphide concentration occurred when the container was fumigated with Degesch Fumi-Cel plates for 96 hours. But, when Phostoxin Prepacs were used to fumigate the container, the buildup of hydrogen phosphide was not as high through the 96 hours. High winds that occurred during this test may account for the low readings. Also, concentrations with this formulation were higher at 24, 48, and 72 hours after fumigation began, before in-transit tests started (table 6), than they were at the same intervals in the static tests (table 5). It is important to note here that all three treatments produced 100% mortality in test insects (see "Bioassays" below).

When the container was fumigated with Detia Gas Ex-B bags and taken on three road trips of 60 miles each, the amount of gas loss could be related to how long after treatment the trip was made (table 6). When the trip began after 24 hours, an average of 66% of the gas was lost; when it began after 48 hours, 34% of the gas was lost. When the trip started after 72 hours, 49% of the gas was lost even though the concentration was low at the start of the trip. This information suggests that the hydrogen phosphide was not evolving rapidly enough from this formulation after 24 hours to make up for losses caused by leaks in transit.

When the Phostoxin Prepacs were used as a source of hydrogen phosphide, 53% of the gas was lost in a 60-mile trip begun 72 hours after fumigant introduction; 75% losses occurred when the container traveled 157 miles 24 hours after introduction, and 78% losses occurred over 156 miles 48 hours after introduction. The similarity of the 24- and 48-hour losses indicates that gas loss with this formulation depends on trip length, not on time after introduction (at least in the 24- to 72-hour period).

The Degesch Fumi-Cel plates produced higher initial concentrations of hydrogen phosphide in the container than did the other formulations (table 6). This was also true in the static tests (table 5). As with the Phostoxin Prepacs, gas loss from the container increased when the length of the trip increased. A 59-mile trip 48 hours after the container was fumigated with this formulation resulted in a 37% gas loss, while three trips from 124 to 130 miles each resulted in 76% to 79% hydrogen phosphide loss. These three trips with similar gas loss rates began either 24, 48, or 72 hours after initial treatment; so, with this formulation, the time of movement of the container was not as important as the length of the trip.

In the one test where the container was held for 2 weeks after hydrogen phosphide introduction (Fumi-Cel plate), including 12 days after a 125-mile trip, the concentration of hydrogen phosphide in the container averaged 69 p/m after the trip (table 6). One week after the initial fumigation, the average concentration of hydrogen phosphide in the container was 30 p/m; 9 days after introduction, the concentration averaged 3 p/m. After 2 weeks, the concentration had dropped to an average of 0.2 p/m.

It is interesting to note that, despite losses of hydrogen phosphide ranging from 34% to 79% from the three formulations used in the 10 in-transit tests, the amount of hydrogen phosphide lost per mile ranged only from 0.8 to 2.1 p/m per mile of travel (table 6). There is no correlation between the percentage of total hydrogen phos-

Table 6.—Concentrations of PH_3 (from each of 3 sources) in a loaded 20-ft container before and after transit

Start of trip (hours after PH_3 introduction)	Miles traveled	Concentration[1] (p/m)		PH_3 loss	
		Start	Finish	Percent	Per mile (p/m)
Detia Gas Ex–B					
24	60	140	47	66	1.6
48	60	256	170	34	1.4
72	60	95	48	49	.8
Phostoxin Prepacs					
24	157	204	50	75	1.0
48	156	180	40	78	.9
72	60	165	78	53	1.5
Degesch Fumi-Cel					
24	124	337	82	76	2.1
48	59	304	192	37	1.9
48	125	325	69	79	2.1
72	130	220	50	77	1.3

[1]Average from 5 sampling lines at different locations in container.

phide lost per trip and the amount of hydrogen phosphide lost per mile.

Gas samples taken in the cab during travel in these tests were all negative for hydrogen phosphide as were all samples taken from the closed cab after break periods. Gas samples taken around the 20-ft container after it was fumigated and sealed were all negative for hydrogen phosphide.

Bioassays

In one static fumigation of the loaded 40-ft container with the floor covered with polyethylene film, we observed an average concentration of 67 p/m of hydrogen phosphide (range: 50 to 250 p/m) 1 day after treatment; 2 days after treatment, the concentration averaged 233 p/m (range: 120 to 350 p/m); 3 days after treatment, the average concentration was 198 p/m (range: 50 to 300 p/m). In the treated container, all bioassay insects were dead after this exposure, while mortality was <2% for the insects in the control container. The hydrogen phosphide concentrations in the 40-ft container for exposures of 71, 91, and 95 hours (table 2) produced 100% mortality of all species at all bioassay sites in the container. High control mortalities (>30.0%) occurred in the almond moth larvae, presumably because of handling or excessive heat.

The mean hydrogen phosphide concentration in the 7-day exposure in the 40-ft container was 155 p/m after 24 hours, 229 p/m after 48 hours, 194 p/m after 72 hours, and 6.0 p/m after 168 hours. This exposure also resulted in 100% mortality of all exposed species and life stages. Control mortality was <2% in all species and life stages.

In the 4-day bioassay studies in the 20-ft container, mortality in treated insects in all exposures (tables 5 and 6) was 100%. Control mortality was <3.4% in all tests except one, where, for unknown reasons, mortality was >90% in both species. Temperatures in the container in these tests ranged from 67° to 107° F when exposures were made to hydrogen phosphide derived from Detia Gas Ex–B bags, from 75° to 107° F when exposures were made to Degesch Fumi-Cel plates, and from 63° to 102° F when exposures were made to hydrogen phosphide derived from Phostoxin Prepacs.

Accident Simulations

Accident simulations with the empty 40-ft container (table 7) showed small amounts of hydrogen phosphide present ($\leq$2 p/m) 1 ft from the doors

Table 7.—Concentrations of PH_3 during accident simulations with 35- and 40-ft containers

Concentration in container (p/m)[1]	Sampling begun (minutes after doors opened)	Concentration (p/m) at— 1 ft from rear of doors	4 ft from rear of doors	Side of doors	Remarks
		40-ft empty container			
60	1	2	0.05	0	Formulation residue still present 96 hours after introduction.
	10	1	0	0	Formulation residue removed.
140	1	.15	.03	0	Formulation residue still present 168 hours after introduction.
160	2.5	1	.005	0	Formulation residue still present 36 hours after introduction.
		40-ft loaded container			
237	1	5	0	0.25	Formulation residue still present 95 hours after introduction.
216	1	12.5	2.5	0	Formulation residue still present 91 hours after introduction.
318	1	10	2.5	0	Formulation residue still present 71 hours after introduction.
	5	2.5	0	0	Formulation residue still present.
		35-ft empty container			
43	1	0	0	0	Curtain in position; formulation residue still present 168 hours after introduction.
331	1	0	0	0	Curtain in position; formulation residue still present 46 hours after introduction.
	10	150	50	25	Curtain removed; formulation residue still present.
	17	60	0	0	Formulation residue still present.
	35	4	1	0	Formulation residue present for the first 15 minutes.

[1]Average from 4 sampling lines at different locations in container.

Table 8.—Concentrations (p/m) of PH_3 (from each of 3 sources) during accident simulations with a loaded 20-ft container

In container[1]	1 ft from rear of doors	3 ft from rear of doors
Detia Gas Ex–B		
30	15.0	0.03
51	.6	.0
86	.3	.0
Degesch Fumi-Cel		
50	2.5	0.0
78	1.0	.0
126	12.5	.3
Phostoxin Prepac		
30	0.1	0.0
54	1.5	.0
56	1.0	.03

[1]Average from 5 sampling lines at different locations in container.

when concentrations were as high as 160 p/m in the container. When tests were run with a loaded 40-ft container, hydrogen phosphide concentrations reached 12.5 p/m 1 ft from the open doors when the mean concentration was 216 p/m in the container.

Tests with the 35-ft container showed no hydrogen phosphide concentrations outside the opened doors when the polyethylene curtain was in place. But, when the curtain was removed, a concentration of 150 p/m was found 1 ft from the opened doors. Removing the hydrogen phosphide residue (unreacted Prepacs, etc.) in this test and leaving the doors open for 18 additional minutes resulted in a drop in concentration to 4 p/m 1 ft from the doors.

For the simulations done in the loaded 20-ft container (table 8) with the Detia Gas Ex–B bags, the highest concentration (15 p/m) of hydrogen phosphide was found with the container having the lowest inside mean concentration (30 p/m). But, in studies made with the Degesch Fumi-Cel formulation, the highest concentration (12.5 p/m) occurred outside the doors of the container having the highest internal mean hydrogen phosphide concentration (126 p/m). All samples taken outside the doors of containers fumigated with Phostoxin Prepacs were 1.5 p/m or less. Samples taken 3 ft from the doors in all tests never exceeded 0.3 p/m.

Aeration Studies

In testing simple aeration (with doors open only, no fans used) of the containers (table 9), we used aeration periods of from 3.5 to 4 hours. When Detia Gas Ex–B bags were used as a source for hydrogen phosphide, 3.5 to 4 hours of aeration produced 85% to 90% reduction in the concentration in the container. In the three tests where Degesch Fumi-Cel plates were used then removed when the doors were opened, there was a 74% to 82% reduction in the hydrogen phosphide concentration in the container. When a plate was used but not removed when the doors were opened, there was only a 64% reduction in the mean concentration of hydrogen phosphide in the container after the doors were opened.

Concentrations of hydrogen phosphide in the container were still too high for safe unloading (more than 0.3 p/m) after aeration when preaeration concentrations were 5.8 p/m or more in these studies (table 9). Fans could be used for more rapid aeration of higher concentrations.

Residue Analyses

Residue analyses showed that none of the treated samples of cornflakes, grits, or rice exceeded the 0.01-p/m legal tolerance established by EPA for processed products. In most cases, the residue on these treated commodities did not exceed the control-residue computations. Of the 26 flour samples examined for residue, 3 exceeded 0.01 p/m. All three samples were in the forward box, away from the hydrogen phosphide source. Residue in the first sample measured 0.011 p/m; the control was analyzed at 0.0014 p/m; and the rear sample had a residue of 0.0056 p/m. This sample was in the container that was fumigated with Detia Gas Ex–B bags and then traveled 60 miles 48 hours after treatment. The second and third flour samples contained 0.011 and 0.015 p/m, while the controls from these tests contained 0.0014 and 0.0013 p/m. These flour samples were from the container that was fumigated with Degesch Fumi-Cel plates and then was either not moved (static fumigation) or traveled 59 miles 48 hours after fumigation.

All samples of cornmeal, including controls,

Table 9.—Concentrations of PH_3 (from each of 2 sources) in a loaded 20-ft container before and after aeration

Temperature (°F)	Windspeed (mi/h)	Concentration (p/m) before doors were opened		Aeration period (hours)	Concentration (p/m) after aeration period	
		Average[1]	Range		Average[1]	Range
Detia Gas Ex-B						
80	0	82	10-150	3.5	12	5-20
70	0	5.8	1-10	3.75	.6	0-1
75	3-5	1.8	1-5	4	.3	0-1
Degesch Fumi-Cel						
95	0	140	100-150	3.5	33	25-50
88	3	69	30-90	3.75	18	5-25
92	5-10	136	100-175	4	24	12-30
85	3-5	50	45-55	[2]4	18	0.5-25

[1]Average of 5 sampling lines at different locations in container.
[2]Fumi-Cel plate not removed during aeration.

had hydrogen phosphide residues greater than 0.01 p/m. In some instances, control sample residues were higher than treated sample residues; so this commodity had probably been previously treated with hydrogen phosphide.

Residue analysis of the blackeyed peas produced highly variable results. In 2 of the 13 fumigations, the residue in the controls and the treatments did not exceed 0.01 p/m. In two other fumigations, both the treatment and control residues exceeded 0.01 p/m. In the remaining nine fumigations, eight of the controls produced residues of 0.0014 p/m, and the ninth had a residue of 0.0083 p/m. The treated blackeyed peas in these fumigations exhibited residues ranging from 0.010 to 0.017 p/m (front samples) and from 0.0049 to 0.076 p/m (rear samples).

CONCLUSIONS

These data show that, under the conditions of these tests, in-transit fumigation of truck-ship containers can be done safely and effectively. Simple sealing techniques were developed for doors and tongue-and-groove floors so that concentrations in the containers were attained initially and maintained during transit to control the insect species and life stages tested. Four formulations producing hydrogen phosphide were tested; three of these were studied in depth for this use. All four provided lethal concentrations during the tests.

These tests also showed that there is no serious danger involved in this form of treatment from an accident since concentrations were always very far below the explosive limit of 17,900 p/m for hydrogen phosphide. Residue analysis from several commodities showed that, in general, the tolerance of 0.01 p/m was not exceeded when the fumigant was used at or slightly below the recommended dosages.

REFERENCE

Rosebrook, D.
1972. Evaluation of phosphine preparation Detia Gas Ex-B. Midwest Res. Inst. Rep. 3502-C, pp. 15-17. Midwest Research Institute, Kansas City, Mo.

APPENDIX.—MATERIALS TESTED FOR SEALING CRACKS IN CONTAINERS HAVING TONGUE-AND-GROOVE FLOORS

Sealant name	Source	Remarks
Adhesive animal glue	GSA 8040-00-290-6273	Thin; runs through cracks.
Cocoon	Diversified Corp. Camden, N.J.	Goes into cracks but will not dry solid even after several coats.
Crack-Stop	Sears Roebuck & Co. Chicago, Ill.	Too thin; runs through cracks.
Dolfinite	Dolphin Paint & Chemical Co. Toledo, Ohio (GSA 8030-00-579-8890).	Too thick; dries slowly.
Fibered roof coating	Gibson-Homans Co. Cleveland, N.J.	Goes into cracks well with brush; does not hold, after 1 or 2 days dries and pulls away from wood; O.K. for metal.
GYM finish	S. C. Johnson & Son Racine, Wis.	Runs through cracks; will not build up.
Latex ceiling paint	Sears Roebuck & Co. Chicago, Ill.	Too runny.
Marco Liquid Nails	SCM Macco Adhesives Nickliffe, Ohio	Fills cracks; does not pull away after drying; labor intensive.
McGill 1-2-3	Out of business	Dries and pulls loose.
Muresco	Benjamin Moore Co. Montvale, N.J.	Must have additive by Gamma Laboratories; consistency of paint not thick enough.
Nu-Sensation Hy-Build	Republic Powdered Metals Medina, Ohio	Too thin, no improvement when mixed with talc.
Tuff-Kote	Tuff-Kote Co. Woodstock, Ill.	Will not fill cracks, too thick; must be troweled on; labor intensive.
USP polymeric roofing	U.S. Polycoat Skokie, Ill.	Used straight, consistency of water; mixed 3 : 1 with baby powder, does not work.

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